

**Standard Operating Procedure for the
Sampling of Particulate-Phase and
Dissolved-Phase Organic Carbon in
Great Lakes Waters**

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1.0 Scope and Application

This Standard Operating Procedure describes the sampling of Great Lakes Waters for particulate-phase organic carbon (POC) and dissolved-phase organic carbon (DOC). Samples of lake water are collected and passed through a 0.7 μM pore-size glass fiber filter. POC is operationally defined as the mass of organic carbon retained on the filter per unit volume of water, and DOC is the material that passes through the filter.

2.0 Safety and Waste Handling

All applicable safety and waste handling rules are to be followed. These include the proper labeling and disposal of chemical wastes. Over-board discharges of chemical wastes are forbidden. Refer to the GLNPO Safety, Health, and Environmental Compliance Manual for specific rules.

3.0 Summary of Procedure

Great Lakes water samples are collected at pre-determined sampling stations and depths *via* either a submersible pump or Rosette sampler. The water is then filtered under vacuum through ashed 47 mm diameter glass fiber filters in an all-glass filtration apparatus. The samples are acidified during the filtration to remove inorganic carbonates. The POC is retained on the filter and frozen at -10 °C until analysis. The filtrate is collected and promptly analyzed for DOC in a ship-board laboratory.

4.0 Description of Apparatus

Water samples (typically 1-4 liters for open-lake locations) are collected from an over-board pump or Rosette sampler. Ashed glass fiber filters are supported in a commercially-available, all-glass, 350 mL vacuum filtration apparatus. Two filtration apparatuses are attached, side-by-side, to ring stands. Samples are filtered simultaneously in duplicate. Tygon tubing (3/8" ID) is used to connect the filtration flasks to an oil-less vacuum pump. The equipment needed are listed in Table 1.

5.0 Preparation of Filters and Reagents

5.1 Preparation of Filters

5.1.1 Filter preparation should take place as close to the start of the survey as possible.

5.1.2 Filters are to be handled only with stainless steel forceps. Filters that are mishandled after the ashing procedure (5.1.4) should be discarded.

- 5.1.3 47 mm diameter GF/F filters (0.7 μ M pore-size) are placed individually in aluminum foil envelopes, dull side of foil facing inward, with three sides folded closed. The fourth side is left open to allow gases to escape from the envelope during ashing.
- 5.1.4 The filters are stacked in a muffle furnace and ashed for four hours at 450 °C.
- 5.1.5 Upon removal from the muffle furnace, the envelopes are sealed on the fourth side.
- 5.1.6 Fifty envelopes containing individual filters are placed into a Ziplock bag and the bag is labeled with the date and initials of the analyst who prepared the filters.
- 5.2 Preparation of Reagents

A solution of 0.2N HCL is prepared by transferring 17 mL of concentrated HCL (16.1N) to a 1000 mL volumetric flask and diluting to the mark with organic-free, distilled, deionized water (from now on referred to as organic-free water). Transfer the solution to a 1 L Teflon squeeze bottle.

6.0 Filtration Procedure

- 6.1 Using stainless steel forceps, place one 47 mm GF/F filter onto the fitted glass support of the sampling apparatus. Place the glass funnel on top of the filter and secure with the clamp. Label the Great Lake name, station number, sampling depth, and date onto the aluminum foil envelope.
- 6.2 Collect the lake water sub-samples from the submersible pump hose or Rosette sampler. Allow the overboard pump line to flush for 15-30 minutes. Collect the lake water into a 4 liter Cubitainer or four, 1 L Teflon bottles. Rinse the container(s) twice with approximately 1 liter of lake water before collecting the sample. If the lake water is to be collected from the Rosette, rinse the container(s) with only 200-300 mL of lake water to ensure there is enough remaining to establish a significant particulate load on the filter (see section 6.7).
- 6.3 Measure the volume of lake water to be filtered in a graduated cylinder, or mark four, 1 L Teflon bottles at the 1 liter level. Prior to filling, rinse the bottles, or cylinder, twice with approximately 100 mL of lake water.
- 6.4 Connect the vacuum pump to the filtration flask. Pour a measured volume of lake water into the glass filtration funnel. Turn on the vacuum pump. Maintain the vacuum between 5-10 inches of Hg during filtration.
- 6.5 After approximately 300 mL of lake water has been filtered, turn off the vacuum pump. Rinse the 200 mL DOC glass sample bottle several times with filtrate and collect approximately 150 mL of the filtrate. Label the Great Lake name, station number, sampling depth and date onto the DOC bottle. Collect the filtrate before step 6.6.

NOTE: Step 6.6 must be done before all the lake water is filtered to ensure that the distribution of the particles on the filter is not disturbed.

- 6.6 Turn on the vacuum pump, and continue pouring lake water into the funnel until sufficient material has been collected (see section 6.7). Just before the last portion of the lake water has been filtered, squirt approximately 5 mL of 0.2N HCL solution into the funnel.
- 6.7 The volume of lake water required to produce a reliable POC measurement (i.e., an amount of material that is within the analytical instrument's linear range) will vary with lake station location, depth, and time of year. For open-lake, oligotrophic conditions, typically 2-4 liters will provide enough material. For near-shore locations, or meso-eutrophic and eutrophic conditions, lake water volumes in the range of 200-500 mL are typical. A filter that becomes visibly loaded with particles and a flow of water through the filter that drops significantly are evidence that sufficient particulate material has been collected.
- 6.8 After the lake water has been filtered, rinse the sides of the funnel with approximately 20 mL of organic-free water and filter this rinse. Turn off the vacuum pump.
- 6.9 Remove the funnel. Using stainless steel forceps, fold the filter in half and place back it into the labeled aluminum foil envelope. Place groups of foil envelopes in a labeled Ziplock bag and store at -10 °C. Record the Great Lake name, station number, sampling depth, volume filtered, analyst, date, and time of day on the POC/DOC Sampling Log Sheet.
- 6.10 Empty the remaining filtrate from the filtration flask.
- 6.11 Rinse the filtration funnel, fitted glass support, filtration flask, and the container(s) with organic-free water.
- 6.12 Re-assemble the filtration apparatus.
- 6.13 Place aluminum foil covers over the filtration funnels.

7.0 Quality Control

- 7.1 A duplicate sample will be filtered in parallel at least once during the sampling of each Great Lake.
- 7.2 A POC/DOC matrix blank will be collected, in duplicate, at the beginning of each survey of the Great Lakes, and at least once during the sampling of each Great Lake. A TSS matrix blank is collected by filtering 1 liter of organic-free water. A DOC matrix blank consists of the filtrate from a POC matrix blank. The matrix blanks are processed identically to Great Lakes water samples.
- 7.3 A POC field blank will be collected, in duplicate, at the beginning of each survey of the Great Lakes, and at least once during the sampling of each Great Lake. A POC field blank is prepared by taking a filter out of the foil envelope, placing it onto the fitted glass support of a clean filtration apparatus, wetting the filter with organic-free water and assembling the filtration apparatus. The apparatus is disassembled, and the filter is removed and processed in the same manner as a sample. There is no field blank for DOC.
- 7.4 Two trip blanks for POC will be processed after the survey has ended. This is done by placing two

filters in their unopened foil envelopes into the Ziplock bag and processing these filters like samples. There is no DOC trip blank.

- 7.5 DOC samples are analyzed promptly, in a ship-board laboratory, during the course of a survey.
- 7.6 Because POC/DOC are parameters which are ancillary to the determination of hydrophobic organic contaminants (HOCs), the POC/DOC samples during an organics survey are taken simultaneous to the HOC samples. Therefore when a HOC matrix blank, field blank or duplicate sample is collected, a POC/DOC matrix blank, field blank or duplicate sample will also be collected.

Table 1: List of Filtration Equipment

<u>Quantity</u>	<u>Equipment</u>	<u>Source or Equivalent</u>
2	Oil-less Vacuum Pump	Schuco 5711-130
6	Teflon wash bottle	Cole-Parmer N-06052-60
2	350 ml, all-glass Filtration apparatus	Nucleopore
2	Stainless Steel Forceps	
2	Support/ring stand for filtration apparatus	

Miscellaneous (some quantities depend on number of samples)

- 47 mm GF/F filters (0.7 µM pore-size)
 - Cubitainers
 - Tygon tubing (3/8"ID)
 - 200 ml glass bottles for DOC
 - permanent markers
 - Ziplock freezer bags
 - Aluminum foil
- Whatman 1825-47